

## Data Validation Report

TDD No: 09-04-01-0011  
PAN: 001275.0440.01TA  
Site: El Dorado Hills  
Laboratory: Lab/Cor, Inc.  
Reviewer: Denise A. Shepperd, Trillium, Inc.  
Date: January 28, 2005

I. Case Summary

## SAMPLE INFORMATION:

Asbestos Samples: CC2-H6-1CP-100504; CC2-H6-2CP-100504; CC2-L6-11CC-100504; CC2-L6-1CA-100504; CC2-L6-1CC-100504; CC2-L6-2CC-100504; CC2-L6-3CC-100504; CC5-H6-1CP-100604; CC5-H6-2CP-100604; CC5-L6-1CA-100604; CC5-L6-1CB-100604; CC5-L6-2CB-100604; NFB-H2-1FD-100504; NFB-H2-2FD-100504; NFB-H2-3FD-100504; SFBA-H2-1FD-100504; SFBA-H2-2FD-100504; SFBA-H2-3FD-100504; SFBA-L2-1CH-100504; SFBA-L2-2CH-100504; SFBA-L2-3CH-100504; SFBA-L2-4CH-100504; SFBA-L2-5CH-100504; SFBB-H2-11FD-100605; SFBB-H2-1FD-100604; SFBB-H2-21FD-100604; SFBB-H2-2FD-100604; SFBB-H2-3FD-100604; SFBB-L2-13CH-100604; SFBB-L2-1CH-100604; SFBB-L2-2CH-100604; SFBB-L2-3CH-100604; SFBB-L2-4CH-100604; SFBB-L2-5CH-100604; SFBC-H2-11FD-100604; SFBC-H2-1FD-100604; SFBC-H2-2FD-100604; SFBC-H2-3FD-100604; NFB-L2-15CH-100504; NFB-L2-1CH-100504; NFB-L2-2CH-100504; NFB-L2-3CH-100504; NFB-L2-4CH-100504; NFB-L2-5CH-100504; SFBC-L2-1CH-100604; SFBC-L2-2CH-100604; SFBC-L2-3CH-100604; SFBC-L2-4CH-100604; SFBC-L2-5CH-100604

Matrix: 49 Air samples  
Analysis: Asbestos by Transmission Electron Microscopy  
Collection Dates: October 5 and 6, 2004  
Sample Receipt Date: October 9, 2004  
Analysis Date: October 12 through November 22, 2004  
Analytical Method: ISO Method 10312

## FIELD QC:

Field Trip Blanks (TB): None  
Filter Blanks (FB): None  
Equipment Blanks (EB): None  
Background Samples (BG): None  
Field Duplicates (D1): Not Identified

**TABLES:**

- 1A: Analytical Results with Qualifications
- 1B: Data Qualifier Definitions for Inorganic Data Review

**SAMPLING ISSUES:**

Six chain of custody (COC) documents were included in the data package and were properly completed except that only the first of these documents was signed as relinquished and received. Each page of the COC records should be signed, dated, and initialed each and every time custody of the samples represented therein is transferred.

These documents included all of the field samples in the data package, as well as many additional samples.

**VALIDATION PARAMETERS AND COMMENTS:****I. Holding Times, Preservation and Sample Integrity**

This parameter is evaluated to ensure that sample custody is documented from collection through analysis, samples are analyzed within the recommended holding time, and that no alteration in sample content has occurred during sample shipment, handling, and storage.

There is no established holding time or storage condition for asbestos samples.

**II. Calibration**

The analyses of materials of known content ensures that identification and quantitation of analytes will be accurate for all samples. Review of the documentation provided for appropriate calibration determines whether or not the analytical results reported by the laboratory are valid and supported by the data.

The data deliverables for this project were included in multiple data packages. The calibration documentation was provided in a single package associated with all of the site sample data packages.

A letter representing documentation of an NVLAP laboratory site assessment conducted on 11/7/03 was included in the data package. The letter included (dated 5/10/04) indicated that the laboratory met the on-site assessment requirements.

Results and evaluator notes and tables were included for an NISTIR 5351 analysis of an inter-laboratory QC sample. The laboratory's raw data were compiled and assessed by Batta Labs. Analysts were identified by initials and included all of the initials documented with this sample set, except "JH." According to the assessor's notes, the sample included chrysotile fibers and structures and the laboratory's results were within NVLAP and NISTIR 5351 acceptance limits. No raw data were provided for this QC sample.

Results for a New York State Department of Health Environmental Laboratory Approval Program proficiency test, conducted between 9/7/04 and 11/9/04, were included. The proficiency samples included asbestos in air. The laboratory's results were satisfactory for all four of the air sample categories. Actinolite and amosite fiber types were identified and counts were acceptable according to the data sheet. No raw data were provided for this proficiency sample. Upon request, the laboratory provided raw data documenting the identification of actinolite and amosite asbestos on 1/27/05. These data were inserted into the QC data package by the validator.

Acceptable instrument calibration was documented in the data package, including screen and camera magnification, camera length and camera constant, spot size, k-factor, beam dose, EDS sensitivity and peak intensity. No documentation of grid opening size was provided. Documentation was provided in the separate proficiency and calibration data package for October through December, 2004, for both of the instruments used for analysis of samples included in this data package. Analyses of the samples in this data set were performed during this time period.

Based on the fact that the laboratory demonstrated proficiency in the performance evaluation (PE) analyses performed in the third quarter of 2004, and that these PE samples included the two predominant asbestos types detected in this field sample set, no action was taken by the validator. It is recommended however, that supporting data be expanded to include raw data supporting the identification of all asbestos types detected in PE samples and demonstration, wherever possible, of the correct identification (in known reference materials) of all fiber types detected in a field sample set.

### **III. Blanks**

Sample matrices known to be devoid of the analytes of interest (method blanks) are prepared and analyzed with each analytical batch. Evaluation of this parameter ensures that contamination introduced during preparation and analyses is not attributed to the field samples.

Other blanks may be generated in the field or laboratory to ensure that no contamination is introduced during sampling and/or storage.

Blanks required for this project included Filter Blanks and Field Trip Blanks. No Filter Blanks or Field Trip Blanks were included with this sample set.

### **IV. Spiked Samples**

The analytes of interest are added in known concentrations to like-matrix blanks or authentic field samples before preparation. This parameter is evaluated in order to assess the laboratory's ability to preserve and recover the compounds of interest.

The analytical method does not require laboratory spiked sample analyses. It is recommended by the validator that some type of laboratory prepared or purchased spiked analyses be performed with each analytical sample batch.

The project requirements specified that results from the most recent inter-laboratory study would be acceptable as an LCS sample for these data. This requirement was met by the laboratory and reported results for the inter-laboratory study sample were acceptable for all air sample parameters (see Section I).

### **V. Duplicate/Replicate Samples**

Results for duplicate/replicate samples are evaluated to assess the laboratory's precision for the analytes of interest in the applicable sample matrix. For asbestos analyses, duplicate and replicate measurements take the form of a combination of variables which include the preparation of the grid, the choice of grid openings to be analyzed, and the analyst performing the counting and identification of structures.

The laboratory included all of the QC samples from all of the field sample sets in a separate data package under a separate report number.

The two analysts, JH and TM, not represented in the PE sample analyses included with the data packages for this project did perform intra-laboratory replicate and duplicate analyses on associated field samples. Results for these QC analyses for both analysts were within the sample-specific acceptance limits.

The quality assurance project plan (QAPP) requires five types of laboratory duplicate/replicate analyses, each to be performed at a rate of 5% (one for every twenty) of the field samples. Based on 25 field samples reported in the data package, at least one or more of each of these QC sample pairs were required. The laboratory compared the primary asbestos structure count for each of the QC samples prepared and analyzed. Results for all of the duplicate/replicate pair types were evaluated based on 95% confidence limits determined from the original sample count result. Results for all of the reported QC samples were within the laboratory's calculated limits. A summary of the laboratory QC samples included with this data set are as follows:

Replicate analyses:

- one sample, CC5-L6-1CA-100604, was analyzed as a replicate wherein a different preparation was analyzed by the same analyst;

Duplicate analyses:

- two samples, CC2-L6-1CA-100504 and SFBC-H2-1FD-100604, were analyzed as duplicates wherein the same grid openings were recounted by a different analyst; and
- one sample, CC5-L6-1CA-100604, was analyzed as a duplicate wherein different grid openings were counted by a different analyst.

No samples were analyzed as QC samples for two of the required categories:

- a replicate wherein different grid openings were selected by the same analyst for a second measurement
- a duplicate wherein a new preparation is analyzed by a different analyst

One or more QC samples should have been included for each of these five QC sample categories in order to satisfy the 5% requirements of the QAPP.

An additional type of QC sample, not identified by the QAPP, was included. Sample CC2-H6-1CP-100504 was recounted by the same analyst, counting the same grids.

According to the QAPP provided with the data packages, field duplicates were required at a rate of 10% of field samples. Field duplicate pairs were not identified or evaluated as part of this validation effort.

## VI. Identification

Identification of asbestos structures and fibers is dependent on sample preparation techniques, analyst training, instrument operation, and data interpretation. Comparison with results from known standards is used to evaluate the accuracy of the structure identification for field samples.

Chrysotile, actinolite, edenite, richterite, winchite, and tremolite were reported by the laboratory in the field samples. According to the report forms provided in the separate QC package, the laboratory correctly identified actinolite, chrysotile, and amosite in PE sample analyses performed in the third quarter of 2004. Comparison of identification between the various analysts, grid opening, and preparations combinations that make up the daily QC for these analyses were acceptable. Therefore; based on the documentation provided, fiber and structure identifications for chrysotile and actinolite were determined to be valid as reported. It was assumed that the laboratory correctly identified the other amphibole structures that were reported in the field samples.

One fiber identified as actinolite on the count sheet for sample SFBC-H2-3FD-100604 was categorized as “other amphibole structure.” No raw data were provided for this fiber, only the measurements listed on the count sheet were available. The basis for this category assignment was not apparent to the validator, however, the ID column indicated “ODQ” indicating that the identification was confirmed by SAED and EDXA. At the data user’s request, the laboratory may be requested to provide supporting documentation for the categorization of this fiber.

## **VII. Quantitation and Reported Detection Limits**

Raw data documentation is reviewed to ensure that all reported results and detection limits are correctly calculated, accurately reported, and supported by the raw data.

With two exceptions, results for asbestos categories, fiber density, and detection limits were correctly calculated and accurately reported by the laboratory. Results were verified by the validator using the information included on the reporting forms and the chain of custody records.

For samples SFBC-H2-1FD-100604 and SFBC-L2-2CH-100604, the entry on the count sheet for the number of grid openings counted did not match the count sheet data. For SFBC-H2-1FD-100604 the count sheet listed 23 grid openings, however the number on the sample final report was 22. For SFBC-L2-2CH-100604, the count sheet showed 50 grid openings counted and the final report number was 49. The validator recalculated the concentrations, analytical sensitivity, and detection limits for these two samples and made the appropriate corrections to the final reports in the data package. These corrections are also reflected in Table 1A of this report, as well as the electronic databases supplied with the data package.

Results from the analyses of 22 field samples (CC2-L6-11CC-100504, CC2-L6-1CC-100504, CC2-L6-2CC-100504, CC2-L6-3CC-100504, CC5-H6-1CP-100604, CC5-H6-2CP-100604, CC5-L6-2CB-100604, NFB-H2-3FD-100504, SFBA-L2-1CH-100504, SFBA-L2-2CH-100504, SFBB-H2-1FD-100604, SFBB-H2-2FD-100604, SFBB-L2-2CH-100604, SFBC-H2-2FD-100604, NFB-L2-15CH-100504, NFB-L2-1CH-100504, NFB-L2-4CH-100504, NFB-L2-5CH-100504, SFBC-L2-1CH-100604, SFBC-L2-3CH-100604, SFBC-L2-4CH-100604, SFBC-L2-5CH-100604) were rejected by the laboratory due to overloading of the filters and were not reported with this data set.

Two samples (CC2-H6-2CP-100504 and SFBA-H2-1FD-100504) were rejected at sample prep because the filters were blown out.

Two of the sample analyses (SFBC-H2-3FD-100604 and SFBC-L2-2CH-100604) were borderline for overloading. Results for these two samples were reported in the data package with total counts of 39 and 102, respectively.

## **VIII. System Performance**

This parameter is evaluated to ensure that the laboratory analytical systems were functioning properly at the time of analyses and that methodology appropriate to the analyses were followed.

The analytical systems appear to have been working satisfactorily and to have been calibrated properly at the time of these analyses, based on the available documentation.

## **IX. Documentation**

Data and documentation completeness is critical in providing support for the reported results. Problems encountered with the nature or quality of the data package documentation are addressed.

No raw data were provided in the data package for the proficiency samples analyzed in support of the laboratory's accreditation. Raw data to support the identification of actinolite and amosite were received upon request on 1/26/05.

Raw data for chrysotile fibers were not included in the data package for review. Raw data documenting fiber identification for the other asbestos types identified in the field samples were present in the data package. Upon request, negatives and EDS for selected samples were received from the laboratory on 1/27/05.

Count sheets included in the data package are computer generated forms. No date of the actual count is presented on these forms. If there is a corresponding bench sheet from which these forms are prepared, these should be supplied as a part of the data package. It is recommended that analyst's initials and date of count be added to the documentation.

The legend for the count sheets, which defines the codes used for the structure counts, lists PSCH as the code for protocol chrysotile structures. The code appearing on the count sheets for this category is PCAS.

On the printouts for the EDS for some of the field samples the analysis date listed is Jan 1, 1997.

Raw data are an integral part of a complete and defensible data package. Edits made on all data should be performed correctly. Proper editing requires drawing a single line through the incorrect information, adding the correct information, and initialing and dating the changes.

Asbestos structures identified in the field samples included actinolite, chrysotile, edenite, winchite, richterite, and tremolite. Examples of known materials included in the data package in support of the sample analyses included only actinolite, chrysotile, and amosite, identified in the proficiency sample analyses. Based on the data provided for validation, the identification of the other fiber types in a known standard was not documented.

#### **COMMENTS:**

- A. Number of grid openings counted, the analytical sensitivity and the concentration was corrected by the validator for samples SFBC-H2-1FD-100604 and SFBC-L2-2CH-100604. Note that in Table 1A the validator has entered the adjusted detection limit for these samples. The laboratory's detection limit is not listed in the tables, instead, the analytical sensitivity is given.
- B. One actinolite fiber in sample SFBC-H2-3FD-100604 was categorized as other "other amphibole structure." From the raw data available the rationale for this decision was not discernible.

#### **ADDITIONAL COMMENTS:**

Results for samples in this data set were determined to be valid as reported by the laboratory with the exceptions noted above. No qualifiers were added to the results by the validator. Reported results, analytical sensitivity, and detection limits are considered to be accurate within the bounds of the 95% confidence limits determined for each sample.

The data results tables included as Table 1A include only the primary and total asbestos structure counts. Counts for individual categories required by the project Scope of Work are presented in the associated electronic data deliverables (EDD) tables.

This report was prepared according to the specifications of the analytical method, ISO Method 10312 "Ambient air - Determination of asbestos fibres - Direct-transfer transmission electron microscopy method," the document



“USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review,” 2/94, and Trillium, Inc.’s SOP No. 0497-06A, for Validation of Analytical Data: Inorganic Analytes.

**TABLE 1B**

**DATA QUALIFIER DEFINITIONS FOR INORGANIC DATA REVIEW**

The definitions of the following qualifiers are prepared in accordance with the document, "USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review," 2/94.

- U    The analyte was analyzed for, but was not detected above the level of the reported value. The reported value is either the sample quantitation limit or the sample detection limit.
- L    Indicates results which fall between the sample detection limit and the CRDL. Results are estimated and are considered qualitatively acceptable but quantitatively unreliable due to uncertainties in the analytical precision near the limit of detection.
- J    The associated value is an estimated quantity. The analyte was analyzed for and was positively identified, but the reported numerical value may not be consistent with the amount actually present in the environmental sample.
- R    The data are unusable. The analyte was analyzed for, but the presence or absence of the analyte cannot be verified.
- UJ   A combination of the "U" and "J" qualifier. The analyte was analyzed for but was not detected. The reported value is an estimate and may be inaccurate or imprecise.